# This Page Is Inserted by IFW Operations and is not a part of the Official Record

# **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

# IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents will not correct images, please do not report the images to the Image Problem Mailbox.

Cross Reference to
Related Application COMPRESSOR LUBRICANT COMPOSITIONS
Field of the Invention (and paragraph)

20

25

30

35

45

The invention relates to lubricant compositions for use in gas compressors, especially sliding-vane rotary compressors.

Background of the Invention
 It is necessary to compress air, carbon dioxide, hydrogen, helium and hydrocarbon gases either for direct use or for transport either in tanks or through pipelines in many applications. Such hydrocarbon gases include compressed natural gas, landfill gas, biogas, digester gas and wellhead gas. The compressors used in such applications, in having moving parts, require lubrication to reduce friction and wear and to provide, in some designs, a sealing effect. Many of the gases, depending upon their sources, contain significant quantities of impurities that can lead to aggressive environments within which the compressors have to work. For example, hydrocarbon gases frequently contain up to 20% of hydrogen sulphide and/or up to 50% carbon dioxide.

Compressors used to compress gases include screw, reciprocating, scroll and sliding-vane rotary compressors. Lubricants used to lubricate such compressors include mineral oils, white oil, poly  $\alpha$  olefins (PAOs) and polyalkyleneglycols (PAGs). Although synthetic lubricants such as PAGs have been used successfully in screw and reciprocating compressors, their use in scroll and sliding vane rotary compressors have not been successful. This is probably due to the higher loads experienced in such compressors, especially sliding-vane rotary compressors in which high loads are experienced at the tips of the vanes and especially along the sides of the vanes as they reciprocate in their guide slots. These problems are exacerbated by acidic impurities in hydrocarbon gases causing corrosion.

In respect of sliding-vane rotary compressors, the lubricant has been typically a mineral oil. However, owing to dilution of the lubricant by the gas being compressed, especially in the case of hydrocarbon gases, the effectiveness of lubrication is limited and the compressor life is relatively short, e.g. 2000 hours. An alternative lubricant that has been used, but only with certain hydrocarbons, is a poly  $\alpha$  olefin lubricant

Clearly, logistically it would be advantageous to have a lubricant composition capable of being used in compressors for a variety of gas applications.

It is an object of the present invention to provide a lubricant composition suitable for use in sliding-vane rotary compressors.

According to the present invention, a lubricant composition for use in a sliding-vane rotary vane compressor comprises:-

- a polyalkyleneglycol base oil component, said polyalkyleneglycol comprising a random copolymer of ethylene oxide (EO) and propylene oxide (PO) having an EO:PO ratio between 3:1 and 1:3 and having been initiated with a compound having five carbon atoms or less;
- b) 0.01% to 10% based on total weight of the composition of an antiwear additive;
- 50 c) 0.05% to 5% based on total weight the composition of an antioxidant;
  - d) 0% to 1% based on total weight the composition of a metal passivator;
- e) 0% to 2% based on total weight the composition of an anticorrosion agent; 55 and

f) 0% to 2% based on total weight the composition of a vapour phase anticorrosion agent.

-2-

- Polyalkyleneglycols and their preparation are described in Synthetic Lubricants and High-Performance Functional 2<sup>nd</sup> Edition Edited by Leslie R Rudnick and Ronald L Shubkin, 1999, 0-8247-0194-1). Particular reference is made to Part I, Section 6 of that publication.
- 10 Preferred polyalkyleneglycols according to the invention have a molecular weight such that the kinematic viscosity of the polyalkyleneglycol is at least 10 cSt, more preferably 12 cSt, at the operating temperatures and pressures of the compressor. In particular, preferred polyalkyleneglycols according to the invention have a kinematic viscosity of at least 10 cSt, more preferably 12 cSt at 100°C.

Preferred polyalkyleneglycols according to the invention have an EO:PO ratio between 2:1 and 1:2, more preferably between 1.5:1 and 1:1.5, but especially 1:1. Furthermore, preferred polyalkyleneglycols according to the invention have been initiated with methanol or butanol.

Preferred polyalkyleneglycols have a viscosity index of at least 150 and, more especially, at least 200. Preferred compositions according to the invention do not have a viscosity improver present therein.

25 Preferred polyalkyleneglycols have a pour point of less than -10°C more preferably less than -20°C and particularly less than -30°C.

20

35

45

50

Preferred polyalkyleneglycols have an acid number of less than 0.2 mgKOH/g.

Preferably, the lubricant composition comprises 0.1% to 5%, more especially 0.5% to 2.5%, based on total weight of the composition of the antiwear additive.

Preferred antiwear additives are selected from phosphates, phosphites, thiophosphates, thiophosphites, dithiocarbomates, amine phosphates and amine phosphates and mixtures thereof. Examples of suitable antiwear additives include tricresyl phosphate, aliphatic amine salt of phosphoric acid monohexyl ester, tri iso nonyl phenyl phosphite and triphenyl phosphorothionate.

Preferably, the lubricant composition comprises 0.5% to 2.5% based on total weight the composition of the antioxidant.

Preferred antioxidants are selected from high temperature antioxidants, for example ashless aminic antioxidants alkylated phenyl napthylamine, alkylated diphenyl amine, polymerized hydroxyquinolines, iminodibenzyl and medium temperature antioxidants, for example gallates, sterically hindered phenolic and diphenolic antioxidants, and mixtures thereof. Examples of suitable high temperature antioxidants include p,p-dioctyldiphenylamine, octyl phenyl napthylamine, polymerised 1,2-dihydro-2,2,4-trimetylquinoline. Examples of suitable medium temperature antioxidants include 6-t-butylphenol, 2,6-dibutylphenol and 4-methyl-2,6-di-t-butylphenol, 2,6-di-t-butyl-alphadimethylamino-p-cresol, propyl gallate and 4,4'-methylene-bis(1,1-dimethyl-ethyl)-phenol.

Metal passivators when present in the lubricant composition to protect metal surfaces exposed to the gases that are being compressed. The metals used in the construc-

-3-

tion of compressors include copper and white metals, eg zinc, aluminum etc, and alloys thereof and other metal alloys including lead-containing alloys.

Preferably, the lubricant composition comprises 0.05% to 0.5% based on total weight the composition of a metal passivator. Examples of suitable metal passivators include gallates, imidazole, benzimidazole, pyrazole, benzotriazole, tolutriazole, 2-methyl benzimidazole, 3,5-dimethyl pyrazole and methylene bisbenzotriazole and mixtures thereof.

5

40

50

Preferably, in the absence of other measures, such as material selection, to prevent corrosion, the lubricant composition comprises 0.1% to 2%, more especially 0.1% to 0.5%, based on total weight the composition of an ashless anticorrosion additive. Example of suitable ashless anticorrosion additives includes amine napthalene sulphonates, amine phosphates, alkenyl succinic half ester, organic poycarboxylic acids and mixtures thereof. In particular ethylene diamine dinonylnapthalene sulphonate, diethylenetriamine dinonylnapthalene sulphonate andaliphatic amine salt of phosphoric acid monohexyl ester and mixtures thereof.

Preferably, in the absence of other measures, such as material selection, to prevent corrosion, and particularly for gas applications that contain acidic impurities (sour 20 gas) the lubricant composition comprises 0.05% to 2%, more especially 0.1% to 0.5%, based on total weight the composition of a vapourphase anticorrosion additive. Although parts of the compressor are submerged under the lubricant composition, there are parts of the compressor and associated pipe-work etc that are ex-25 posed to the gases and any aggressive impurities they carry. Vapour-phase anticorrosion agents are volatilised from the lubricant composition at the operating temperatures of the compressor and coat other exposed surfaces to protect them from attack. Examples of suitable vapour-phase anticorrosion agents include dicarboxylic acids, silicones, siloxanes, silanes, silicates and volatile amines and mixtures thereof. In particular, the dicarboxylic acids are C7 or higher acids for example docecanedioc 30 acid; and the Si-containing compounds include decamethycyclopentasiloxane, dimethylsiloxane pentamer, trimethylsilyl (2,6-di(trimethylsiloxy)phenyl) methanoate, triethoxy (3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl) silane and tetraethyl silicate; and volatile amines include primary amines, tripropylamine and ethyl-di-2-35 ethylhexylamine.

Lubricant compositions according to the invention may also comprise one or more other lubricant additives of known functionality at levels between 0.0001 and 20 %, more preferably between 0.01 and 10% more especially between 0.01 and 5%. Suitable additives include extreme pressure agents, acid scavengers, foaming agents, anti-foaming agents, stabilisers, surfactants, lubricity improvers or oiliness agents and friction modifiers.

According to another aspect of the invention, the use in a sliding-vane rotary vane compressor of a lubricant composition comprising:-

- a) a polyalkyleneglycol base oil component, said polyalkyleneglycol comprising a random copolymer of ethylene oxide (EO) and propylene oxide (PO) having an EO:PO ratio between 3:1 and 1:3 and having been initiated with a compound having five carbon atoms or less;
- b) 0.01% to 10% based on total weight of the composition of an antiwear additive;
- 55 c) 0.05% to 5% based on total weight the composition of an antioxidant;

- d) 0% to 1% based on total weight the composition of a metal passivator:
- e) 0% to 2% based on total weight the composition of an anticorrosion agent; and
  - f) 0% to 2% based on total weight the composition of a vapour-phase anticorrosion agent.
- According to yet another aspect of the invention, a method of lubricating a rotary vane compressor comprises utilising a lubricant composition comprising:-
- a) a polyalkyleneglycol base oil component, said polyalkyleneglycol comprising a random copolymer of ethylene oxide (EO) and propylene oxide (PO) having an EO:PO ratio between 3:1 and 1:3 and having been initiated with a compound having five carbon atoms or less;
  - 0.01% to 10% based on total weight of the composition of an antiwear additive;
    - c) 0.05% to 5% based on total weight the composition of an antioxidant;

20

55

- d) 0% to 1% based on total weight the composition of a metal passivator;
- e) 0% to 2% based on total weight the composition of an anticorrosion agent; and
- f) 0% to 2% based on total weight the composition of a vapour-phase anticorrosion agent

According to a further aspect of the invention, a sliding-vane rotary compressor charged with a lubricant composition comprising:-

- a polyalkyleneglycol base oil component, said polyalkyleneglycol comprising a random copolymer of ethylene oxide (EO) and propylene oxide (PO) having an EO:PO ratio between 3:1 and 1:3 and having been initiated with a compound having five carbon atoms or less;
- 40 b) 0.01% to 10% based on total weight of the composition of an antiwear additive;
  - c) 0.05% to 5% based on total weight the composition of an antioxidant;
- 45 d) 0% to 1% based on total weight the composition of a metal passivator;
  - e) 0% to 2% based on total weight the composition of an anticorrosion agent; and
- 50 f) 0% to 2% based on total weight the composition of a vapour-phase anticorrosion additive.

In a particularly preferred embodiment of the invention, there is provide a lubricant composition for use in a sliding-vane rotary vane compressor comprising:-

a) a polyalkyleneglycol base oil component, said polyalkyleneglycol comprising a random copolymer of ethylene oxide (EO) and propylene oxide (PO) having an EO:PO ratio between 1.5:1 and 1:1.5 and having been initiated by methanol or butanol and having a kinematic viscosity of at least 12 cSt at 100°C;

- b) 0. 1% to 5% based on total weight of the composition of an antiwear additive;
- c) 0.5% to 2.5% based on total weight the composition of an antioxidant;

5

20

35

40

55

- 10 d) 0.1% to 0.5% based on total weight the composition of a metal passivator;
  - e) 0% to 2% based on total weight the composition of an anticorrosion additive; and
- 15 f) 0% to 0.5% based on total weight the composition of a vapour-phase anticorrosion additive.

Preferred lubricant compositions according to the invention consist essentially of said polyalkyleneglycol base oil component and additives.

Preferred lubricant compositions according to the invention have an acid number of less than 0.5 mgKOH/g.

Lubricant compositions according to the invention provide good lubrication in slidingvane rotary compressors with a variety of gases. In particular, the polyalkyleneglycol has a relatively low solubility in the gases but has the ability to absorb water. The low gas solubility ensures, along with the specified additives, that there is sufficient lubricant composition present to lubricate the sliding vanes and their tips and to provide a seal at the vane tips between the high and low pressure sides of each vane.

As sliding-vane compressors may have significant down time, the ability of lubricant compositions according to the invention to absorb water that condenses from the gas as the compressor cools down as compared to any water remaining free in the compressor means that corrosion of metal components by the water is minimised or prevented. Some gases, such as hydrocarbon gases, tend to be particularly wet giving rise to significant quantities of water condensing in the system to accumulate in the compressor sump. The lubricant composition of the invention forms a single phase with water over the normal operating temperature of the compressor. The absorbed water, following start up of the compressor, does not affect the efficacy of the lubricant composition, the single phase of lubricant composition and water being pumped through the system to lubricate it. As the compressor heats up to operating temperature, the absorbed water volatilises out of the lubricant composition and is swept out of the compressor by the gas flow there through.

This is in contrast to mineral oil and PAO lubricants that are immiscible with water and, therefore, allow free water to accumulate in the compressor giving rise to corrosion problems, especially in acidic environments, and to problems of water instead of lubricant being pumped from the sump through the system on start up of the compressor, the water not being effective in lubricating the compressor.

Lubricant compositions according to the invention also have the advantage of working in compressors used for pumping a variety of gases including hydrocarbon gases and air. Thus, it offers major logistical advantages in that compressor manufacturers only need stock one grade of lubricant avoiding issues of separate storage tanks for multiple grades, potentially filling compressors with the wrong lubricant etc.

The low cost of maintenance of sliding-vane compressors make them particularly useful in gas-boosting applications, particularly for micro-turbine applications.

A booster machine compresses air or gas from a pressure above atmospheric to a still higher pressure. Booster machines have many uses, especially in oil and gas fields and related industries. Examples of gas boosting are the feeding of wellhead gas to pipelines or of natural gas to gas turbines. In these latter applications, the compressor is used to supply gas at the flow rate and pressure needed for continuous operation of the turbine. Even small amounts of petroleum-based lubricants carried over in the gas to the turbine may produce carbonaceous deposits in the gas inlet nozzles of the turbine restricting flow and causing flameout. The low carry over, high thermal stability and clean burning capabilities of lubricant compositions according to the invention make them particularly suited for this role. In such applications, the compression of the gas may be either single- or multistage, depending upon the pressure differentials, horsepower, and the analysis of the gas.

The invention will now be described further by way of example only with reference to the accompanying drawings and the following Examples.

Brief
- In the drawings: Description of the Drawings

20

25

35

50

55

Figure 1 shows a diametrical section through of a sliding-vane rotary compressor; and

Figure 2 is a graphical representation of the results obtained in Example 2;

Figure 3 is a graphical representation of the results obtained in Example 3; and

Figure 4 is a graphical representation of the results obtained in Example 5.

Description of the Invention

Referring to Figure 1, the sliding-vane rotary compressor 10 has housing 12 having a cylindrical bore 14. The low-pressure side of the compressor 10 has a gas inlet 16 leading into the bore 14. A high-pressure gas outlet 18 extends from the bore 14 at a location circumferentially remote from the gas inlet 16. A rotor 20 is mounted in the bore 14 for rotation about an axis, the axis being offset from the axis of the bore 14 such that the rotor 20 is in sliding contact with the bore 14 between the outlet 18 and the inlet 16 in the direction of rotation of the rotor 20

The rotor 20 has slots 22 that are equi-circumferentially spaced around its periphery. The slots 22 extend tangentially to a circle centred on the axis of the rotor 20 but of diameter smaller than the rotor 20 whereby the slots 22 are inclined relative to the rotor 20 in the direction of rotation thereof. Each slot 22 has a vane 24 mounted in it; the vanes 24 each being able to slide in its slot 22 under the influence of centrifugal force outwardly relative to the rotor 20 to engage the bore 14.

In operation, the rotation of the rotor 20 causes the vanes 24 to be forced into engagement with the bore 14 and define between adjacent vanes 24 a variable gas compression volume 26. Gas entering the gas compression volumes 26 through the inlet 16 is compressed as the vanes sweep through the bore 14, the volumes 26 decreasing in volume as the vanes 24 approach the outlet 18.

A lubricant composition is present in the compressor 10 to lubricate the sides of the vanes 22 as they slide in the slots 22. The lubricant composition also lubricants and provides a satisfactory seal between the high and low pressure sides each vane 22

# Example 1

Lubricant compositions (Samples 1 and 2) according to the invention has as a base oil component a butanol-initiated polyalkylene glycol having an EO:PO ratio of 1:1 and has the additives shown in Table 1.

Table 1

Sample 1	Sample 2	
0.5%	1%	tricresyl phosphate, an antiwear additive.
0.05%	0.05%	Irgalube 349 available from Ciba-Geigy, an antiwear and anticorrossion additive consisting of aliphatic amine salt of phophoric acid monohexyl ester.
0.3%		Oloa 233FA available from Chevron, a dicarboxylic acid anticorrosion additive
10ppm		Tego 793 available from Goldschmidt, an antifoam additive consisting of a modified poyether-polysiloxane
	0.3%	Irgacor L12, available from Ciba, an alkenyl succinic acid half ester antioxidant additive.
0.5%		Vanlube RD available from Vanderbilt, an antioxidant additive consisting of polymerised 1,2-dihydro-2,2,4-trimetylquinoline
	0.5%	3,5-dibutyl-4-hydroxytoluene, an antioxidant.
0.5%		Ethanox 702 available from Albemarle, an antioxidant additive consisting essentially of 4,4'-methylene-bis(1,1-dimethyl-ethyl)-phenol

All % shown in the table are weight % based on the total composition.

Samples 1 and 2 have the properties shown in Table 2.

### 15 <u>Table 2</u>

10

Properties	Test	Sample 1	Sample 2
Viscosity (cSt) at 40°C	ASTM D-445	83.3	82-84
Viscosity (cSt) at 100°C	ASTM D-445	16.0	
Viscosity Index	ASTM D-2270	206	<del></del>
Pour Point (°C)	ASTM D-97	-38	<u> </u>
Flash Point C)C (°C)	ASTM D-92	261	f
Acid Value (mgKOH/g)	ASTM D-974	0.33	
Copper Corrosion	ASTM D-130/94	Pass – 1A	
Steel Corrosion	ASTM D-665A	Pass -	
•		No cor-	
4.0.11.104		rosion	
4 Ball Wear Scar (mm)	ASTM D-2783	0.73	

#### Example 2

The effect of a hydrocarbon, eg heptane, on the viscosity of Sample 1 and of comparative samples, namely Sample 3 – a commercially available mineral oil formulation used in sliding-vane rotary compressors available under the trade name Hydrovane 2000 from Compair Hydrovane – and Sample 4 – a commercially available

phthalate ester formulation used in sliding-vane rotary compressors available under the trade name Compair CS300 from Compair — was tested. The test was done by first measuring the viscosity of the neat samples. Then the viscosity of the samples following exposure to heptane was measured. The samples were exposed to heptane by pouring 40mls of the sample into a measuring cylinder and adding 4mls, ie 10%, of heptane into the cylinder. The sample and the heptane were stirred together for 5 minutes and then left to separate for one hour. The heptane layer that separated from the sample was then removed and the viscosity of the sample determined. The test was repeated with fresh quantities of the samples and added amounts of heptane at levels of 8mls, ie 20%, 12mls, ie 30%, and 16mls, i.e. 40%.

The viscosity of each of the tested samples was measured using the ASTM D445 method at 40°C.

The results are shown in Table 3 and are shown in graphical form in Figure 2. As can be seen, the viscosity of Sample 1 remains significantly higher at high hydrocarbon loading as compared to the viscosity of the commercially-available Samples 3 and 4 and, consequently, the lubricity effect of Sample 1 will be higher than that of Samples 3 and 4 under those high loading conditions.

Table 3

10

25

30

35

40

Amount of Heptane	Sample 1	Sample 3	Sample 4
0	81	130.52	100.8
10%	36.6	43.9	24.3
20%	18.9	19.5	9.61
30%	18.9	9.91	4.69
40%	18.9	5.79	2.45

## Example 3

The ability of Samples 1 and 3 to resist removal from metal surfaces was by hydrocarbon was tested. In order to determine the comparative resistance of lubricants to be washed off by liquid heptanes a simple laboratory method has been developed. Five numbered steel plates, per sample, are washed thoroughly in white spirit and acetone, and then hung up until dry. Each plate is weighed to four decimal places with out being touched. The clean, dry plates are immersed into the sample, up to a marked line and hung up again. After an hour, they were re-weighed. From the results, the mass of lubricant left on the plates after each wash can be calculated and a graph plotted. The coupon was then dipped into heptane, removed and, after one hour, re-weighed. This was repeated four times. The process was repeated with a fresh coupon-using Sample 2.

The results are shown in Table 4 and are shown in graphical form in Figure 3. As can be seen, the amount of Sample 1 and hence the thickness of the remaining lubricant composition on the coupon is significantly higher than for Sample 3. This is particularly important at start up of a compressor, especially if it has been standing unused for some time. The retention of a film of lubricant composition ensures the surfaces to be lubricated are lubricated and not metal to metal contact.

#### 45 <u>Table 4</u>

No of Times	Sample 1	Sample 3
Dipped	Weight of Oil	Weight of Oil

9-

	on Coupon (g)	on Coupon (g)
0	0.1175	0.1042
1	0.0442	0.0361
2	0.0357	0.0223
3	0.0325	0.0167
4	0.03	0.01
5	0.025	0.005

## Example 4

To check the potential of lubricant compositions for thermal decomposition under operating conditions, equal quantities of Samples 1 and 4 were placed in glass tubes within an aluminium-heating block. A steel coupon (76mm by 13mm by 3mm in size) was placed in each tube containing Samples 1 and 4. The glass tubes were connected via silicone tubing to respective similar tubes containing distilled water that were situated outside the heating block. The function of the water in the absorption tubes was to absorb any decomposition product volatilised from the samples of lubricant composition.

Compressed dry nitrogen was passed in sequence through the tubes containing Samples 1 and 4 and through the respective tubes containing water at a rate of 1 litre/hour. The test was carried out at 175°C for 168 hours.

After the test duration, the samples were tested for kinematic viscosity @ 40 °C and Acid Value (Neutralisation number) and were then compared to the initially-measured values of those parameters for evaluation of the performance of the lubricant compositions. The acid value measurement was a combination of acid values of both the Samples 1 and 4 and the respective water samples associated therewith to allow for the fact that low molecular weight acid from the decomposition of the lubricant compositions were volatilised from the Samples 1 and 4.

#### 25 The results are shown in

#### Table 5

15

20

Lubricant	Sample 1	Sample 4
Viscosity at 40°C In cSt	79.3	100.8
%Change in viscos- ity at end of test	0.9	4.2
Initial Acid Value in mgKOH/g	0.32	0.09
Change in Acid Value in mgKOH/g	0.02	0.49

30 Sample 1 has a much lower change in viscosity and acid value as compared to Sample 4

#### Example 5

Compair-Hydrovane air compressors were charged with lubricant compositions of Samples 1, 3 and 4 and Samples 5 and 6 which were respectively a PAO available from Mobil and phthalate ester formulation used in sliding-vane rotary compressors available under the trade name Compair CS500 from Compair. The compressors

were run continuously apart from being stopped at intervals to allow sampling of the lubricant compositions. The samples of the compositions were analysed for iron content, the amount of iron content being indicative of wear in the compressors. The results are plotted in Figure 4. The normal oil change interval for mineral oil is indicated on the graph as 2000 hours. Although the graph only goes up to 2500 hours, the tests were in fact run for a total of 4000 hours before being stopped.

-10-

As can be seen from Figure 4, Sample 1, in accordance with the invention, performed significantly better than the lubricant compositions normally used for this application.

# Example-5-6

5

10

In field tests using a lubricant composition having a specification based on Sample 1, compressors used in well-head applications, including those involving aggressive (sour) gas compositions, have exceeded 8000 hours service. Similarly, in microturbine applications, compressors have exceeded 10000 hours service.

Lubricant compositions according to the invention permit the lubricant in a compressor sor to be changed at the compressor service intervals, eg one year, rather than at an oil service interval of say 2000 hours that was necessary using existing lubricant compositions.